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6. AUTHOR(S) R. L. Wells, J. F. Janik, V. W. L. Gladfelter, J. L. Coffey, M. A. Johnson, and B. D. Steffey			Dr. Harold E. Guard	
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New Precursor Routes to Nanocrystalline Cubic/Hexagonal Gallium Nitride, GaN

R. L. Wells,¹ J. F. Janik,¹ V. W. L. Gladfelter,² J. L. Coffey,³ M. A. Johnson,³ and B. D. Steffey³

1. Department of Chemistry, Duke University, Durham, NC 27708

2. Department of Chemistry, University of Minnesota, Minneapolis, MN 55455

3. Department of Chemistry, Texas Christian University, Fort Worth, TX 76129

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Duke University
Department of Chemistry,
P. M. Gross Chemical Laboratory
Box 90346
Durham, NC 27708-0346

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NEW PRECURSOR ROUTES TO NANOCRYSTALLINE CUBIC/HEXAGONAL GALLIUM NITRIDE, GaN

R. L. WELLS*, J. F. JANIK‡, W. L. GLADFELTER#, J. L. COFFER†, M. A. JOHNSON†, and B. D. STEFFEY†

* Department of Chemistry, Duke University, Durham, NC 27708-0346

‡ on leave from the University of Mining and Metallurgy, Krakow, Poland

Department of Chemistry, University of Minnesota, Minneapolis, MN 55455

† Department of Chemistry, Texas Christian University, Ft. Worth, TX 76129

ABSTRACT

Two precursor routes culminating in bulk nanocrystalline gallium nitride materials are reported, with emphasis on the materials' XRD/crystalline features and photoluminescence (PL). First, the new polymeric gallium imide, $\{\text{Ga}(\text{NH})_{3/2}\}_n$, can be converted to nanocrystalline, cubic/hexagonal GaN ranging in color from yellow to light gray. Second, a new route to gallazane, $[\text{H}_2\text{GaNH}_2]_x$, from the combination of LiGaH_4 and NH_4X ($\text{X} = \text{Cl}, \text{Br}$) in Et_2O is shown to result in a material that slowly converts to a polymeric solid *via* H_2 and NH_3 elimination-condensation pathways. Both the gallazane and the polymeric solid are pyrolyzed to dark gray nanocrystalline, phase-inhomogeneous GaN as above. Specific variations in the pyrolysis conditions enable some control over the particle nanosize and a degree of crystalline phase-inhomogeneity of the materials. These nanophase GaN materials have also been characterized by room temperature photoluminescence (PL) measurements. In general, the observed emission spectra are strongly dependent on pyrolysis temperature and typically exhibit weak defect yellow-green emission. While the as-prepared GaN does not exhibit band-edge PL, a brief hydrofluoric acid etch yields nanophase GaN exhibiting an intense blue-emitting PL spectrum with an emission maximum near 420 nm.

INTRODUCTION

The broad direct bandgap semiconductor gallium nitride, GaN, has two common crystalline polymorphs - the thermodynamically stable hexagonal or wurzite form and the metastable cubic or zinc blende form [1]. Most of the vapor deposition processes as well as bulk precursor routes result in the hexagonal GaN, a variety compatible with the Group 13 nitrides, AlN or InN, with which it can form solid solutions of intermediate semiconductor/electronic properties.

In recent years, however, there has been an increasing number of reports demonstrating the formation of cubic GaN from precursor routes under standard laboratory conditions. Gladfelter and coworkers [2] reported that the pyrolysis of the cyclotrigallazane, $[\text{H}_2\text{GaNH}_2]_3$, (obtained from the reaction between $\text{H}_3\text{Ga}\cdot\text{NMe}_3$ and NH_3 [3]), resulted in the cubic/hexagonal variety of GaN best described as a mixture of cubic and hexagonal close-packed layers. Wells and Janik [4] found that the conversion of the new gallium imide precursor, $\{\text{Ga}(\text{NH})_{3/2}\}_n$, yielded similar cubic/hexagonal GaN. In a related precursor chemistry, Gonsalves *et al.* [5] described the pyrolysis of $[\text{Ga}(\text{NMe}_2)_3]_2$ under NH_3 which led to GaN labeled as cubic with stacking faults. However, based on model calculations, most of these results can be interpreted as having varying amounts of order in the stacking direction [2b, c]. For the purpose of this report, we will use interchangeably "cubic/hexagonal" or "phase-inhomogeneous" to denote such GaN phases.

In regard to the luminescent spectral behavior of the GaN materials, generally there are four types of possible scenarios anticipated: first - intrinsic bandgap photoluminescence, emitting in the blue region with a maximum near 410 nm, second - broad defect photoluminescence, known to emit in the yellow region [6], third - non-radiative states, lowering the observed quantum yield of band edge and/or defect photoluminescence, and fourth - any possible combination of the above three scenarios.

EXPERIMENTAL

Synthetic Procedures

The preparation of the polymeric gallium imide, $\{\text{Ga}(\text{NH})_{3/2}\}_n$, was accomplished according to the published method [4]. Before use, the imide was conditioned under ammonia for one day.

The gallazane, $[\text{H}_2\text{GaNH}_2]_x$, was synthesized from the new reaction system, $\text{LiGaH}_4/\text{NH}_4\text{X}$ ($\text{X} = \text{Cl}, \text{Br}$) in Et_2O , to be described elsewhere [7]. The optimal preparation used the *in situ* formed LiGaH_4 (not isolated from the ether-liquor) in combination with solid NH_4Cl at 0°C , wherein the ether insoluble LiCl could be conveniently separated from the initially soluble gallazane. If, on the other hand, one isolated LiGaH_4 by evacuation, the compound partially decomposed forming an insoluble gray slurry upon redissolution in fresh Et_2O and contaminating the products. The isolated white gallazane decomposed at room temperature with the evolution of H_2 and NH_3 , and converted slowly to a gray polymeric solid, elimination-condensation product, especially upon evacuation. Also, a several day long stirring of the $\text{LiGaH}_4/\text{NH}_4\text{Br}$ system resulted in the spontaneous precipitation of yet another polymeric solid similar to the one just described. The as-prepared gallazane, as well as the polymeric solids obtained from it after definite reaction and evacuation times, were immediately used in all subsequent pyrolyses. Due to the thermal instability of both the LiGaH_4 and $[\text{H}_2\text{GaNH}_2]_x$, the preparation details (*in situ* or isolated materials) and handling times of the by-products as well as evacuation appeared to influence the properties of the final GaN materials.

For pyrolysis under an ammonia flow, liquid NH_3 over sodium was slowly evaporated into the furnace-heated pyrolysis tube that contained a precursor in a quartz boat, and left the system through a bubbler. A decomposition of the gallium imide was also performed in boiling N,N,N',N' -tetramethyl-1,6-hexanediamine (bp 210°C). In this experiment, an amine suspension of the imide was placed in a flask equipped with a condenser and was refluxed under argon for 48 hours. Upon completion, the supernatant was removed and the solid evacuated overnight yielding a yellow grayish product.

Characterization Methods

XRD data were collected using oil coated samples on a Phillips XRD 3000 diffractometer. Elemental analyses were provided by E + R Microanalytical Laboratory, Corona, NY. Room temperature photoluminescence (PL) data were recorded using a SPEX Fluorolog-2 instrument equipped with a double emission monochromator and a R928 photomultiplier tube. Excitation was provided by a 450 W Xe lamp whose output was focused into a 0.22 m monochromator to provide wavelength selection. PL spectra for some samples were also recorded by excitation with the 325 nm line of a Linconix HeCd laser with an average power of 7 mW.

Organization and Characterization of Samples

The gallium imide derived GaN samples have the following labels for the listed pyrolysis conditions: **A** - reflux in the amine, bp 210°C , 48 h; **B** - 600°C , vacuum, 3 h; **C** - 500°C , NH_3 , 4 h; **D** - 600°C , NH_3 , 4 h. These four samples were used for the XRD determinations. Some of them were also used in the photoluminescence (PL) study. An additional sample was labeled **E** - 300°C , NH_3 , 4 h.

The gallazane derived samples of GaN have the following designations (in parentheses, preparative route to gallazane): **1** - pyrolysis of the polymeric solid/gallazane obtained upon 20 h evacuation of gallazane (*in situ* $\text{LiGaH}_4/\text{NH}_4\text{Br}$) - 600°C , vacuum, 4 h; **2** - pyrolysis of the precipitated (60 h) polymeric solid (evacuated $\text{LiGaH}_4/\text{NH}_4\text{Br}$) - 600°C , NH_3 , 3 h; **3** - pyrolysis of the polymeric solid/gallazane obtained upon 5 h evacuation of gallazane (evacuated $\text{LiGaH}_4/\text{NH}_4\text{Br}$) - 600°C , NH_3 , 3 h; **4** - pyrolysis of the precipitated (72 h) polymeric solid (*in situ* $\text{LiGaH}_4/\text{NH}_4\text{Br}$) - 600°C , NH_3 , 4 h; **5** - pyrolysis of the as-prepared gallazane (*in situ* $\text{LiGaH}_4/\text{NH}_4\text{Cl}$) - 600°C , NH_3 , 5 h. Samples **1-5** were used for the XRD determinations and sample **4** was additionally included in the photoluminescence (PL) measurements.

EA: **C**: Ga, 81.02; N, 15.87; C, 0.22; H, 0.54; Ga/N = 1.03/1.00; **3**: Ga, 83.06; N, 16.54; H, 0.05; C < 0.3; Ga/N = 1.05/1.00; **5**: Ga, 86.31; N, 13.49; C, H < 0.1%; Ga/N = 1.29/1.00.

RESULTS AND DISCUSSION

Two different GaN precursors used in this study, i.e. gallium imide and gallazane (*via* a new synthetic route from $\text{LiGaH}_4/\text{NH}_4\text{X}$ ($\text{X} = \text{Cl}, \text{Br}$) in Et_2O), were pyrolytically converted to a range of similar phase-inhomogeneous GaN materials. The difference between the chemical and physical properties of the product formed from the reaction of LiGaH_4 with NH_4X (designated gallazane) and that of previously characterized cyclotrigallazane is noteworthy. Cyclotrigallazane produced directly from the reaction of $\text{H}_3\text{Ga}\cdot\text{NMe}_3$ and NH_3 is an insoluble, white crystalline solid that has been fully characterized. It can be slowly sublimed under vacuum at 80°C without decomposition, and thermogravimetric analyses established a weight loss only above 140°C . In contrast, the material produced by the double hydrogen elimination from LiGaH_4 plus NH_4X exhibits solubility in ether and a greater thermal instability. This new synthetic route may produce material having the formula $[\text{H}_2\text{GaNH}_2]_x$ where x does not exclusively equal 3. It is notable that some of the syntheses of borazane $[\text{H}_2\text{BNH}_2]_x$ yield a family of compounds related by different values of x .

Figure 1 illustrates some of the XRD spectra obtained for the gallium imide derived materials.

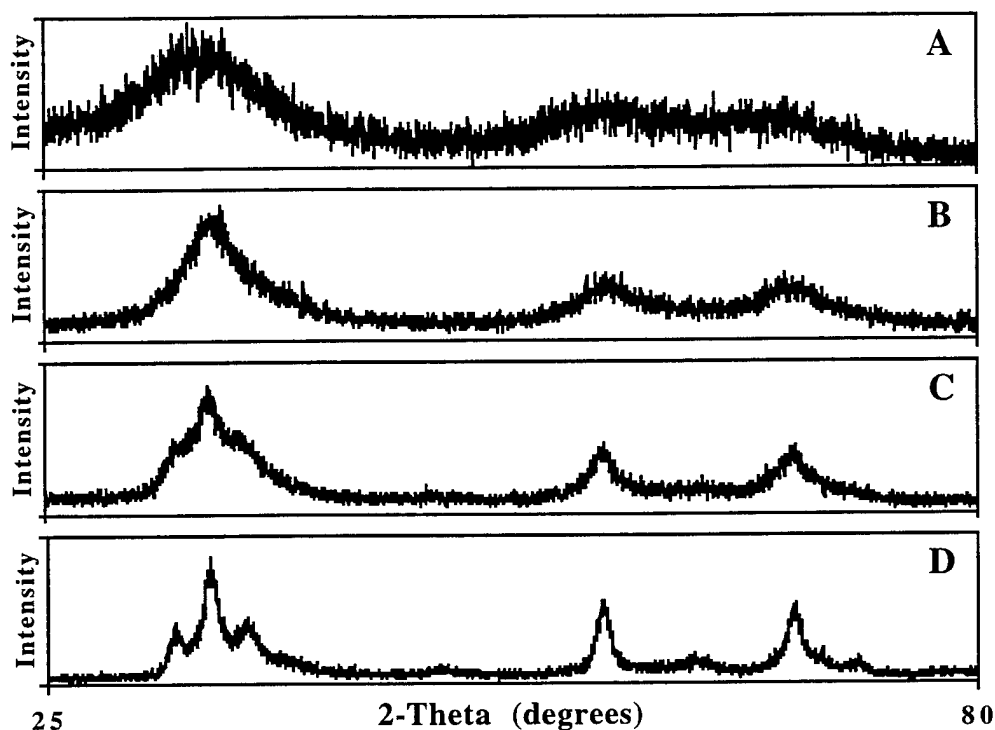


Figure 1. XRD powder patterns for GaN obtained by pyrolysis of gallium imide: **A** - reflux in $\text{N}, \text{N}, \text{N}', \text{N}'$ - tetramethyl - 2, 6 - hexanediamine, bp 210°C , 48 h; **B** - 600°C , vacuum, 3h; **C** - 500°C , NH_3 , 4 h; **D** - 600°C , NH_3 , 4 h.

None of the samples shows a typical diffraction pattern for the hexagonal GaN (JCPDS file 2-1078) with the characteristic triplet feature in the 2-theta 30° to 40° range for the (100), (002), and (101) diffractions at (in parentheses, intensity) 32.4° (70), 34.6° (50), and 37.0° (100), respectively, which, approximately and with caution, can be used to compare with those of the phase-inhomogeneous GaN samples. For example, the spectra for samples **C** and **D** show the triplet nature but with the intensities different from the relevant hexagonal pattern. The high intensity middle peak in the triplet may correspond to either a significant cubic phase in the admixture with some hexagonal phase or the whole pattern may result from statistically scrambled layers of cubic and hexagonal GaN in a certain ratio [2a, b]. Alternatively, the pattern can be accounted for by assuming different quantities of stacking faults in either the cubic or hexagonal varieties, or by different cubic or hexagonal polymorphs [2c]. Although the phase character of

these solids seems to be varied and remains unknown, it is clear that it is not purely hexagonal or cubic but rather of an intermediate and complex origin.

Sample **A** was obtained at the lowest temperature of 210 °C after reflux in the amine. It shows three diffraction halos in the positions that are consistent with the onset of GaN crystallinity. The remaining diffractograms confirm the influence of the applied pyrolysis conditions on both the nanocrystallinity and phase character of the samples. The average crystallite size estimated from the Scherrer equation is 13 nm for sample **D** and smaller for other samples. Samples **B** and **D** were both heated at 600 °C; however, the first one (pyrolyzed under vacuum) displays very broad features and, hence, contains small crystallites while the second one (pyrolyzed under NH₃) is relatively much sharper and consists of bigger crystallites. In addition to the application of vacuum or a NH₃ atmosphere, the pyrolysis temperature is also found to influence the particle nanosize. This is evident from comparison of the patterns for samples **C** and **D** (pyrolysis under NH₃); the increase of the temperature from 500 °C (sample **C**) to 600 °C (sample **D**) results in a noticeable sharpening of the peaks and, thus, increased particle size.

Figure 2 shows the XRD results for the gallazane derived GaN materials. In this case, all the pyrolyses were performed at 600 °C. Similarly, as in the relevant gallium imide route case, the pyrolysis under vacuum yields smaller average nanoparticles than the pyrolysis under NH₃ (compare sample **1** with all the remaining samples). In this case, however, the diffractions are broader than for related sample **B** which implies this precursor route affords smaller nanoparticles of GaN than the gallium imide route under similar pyrolysis conditions. The Scherrer estimation gives for sample **3** the largest in the series average particle size of 13 nm, coinciding well with the particle sizes approximated above for the gallium imide derived GaN. Also, sample **5** obtained from the as-prepared gallazane shows a pattern almost identical with sample **3**.

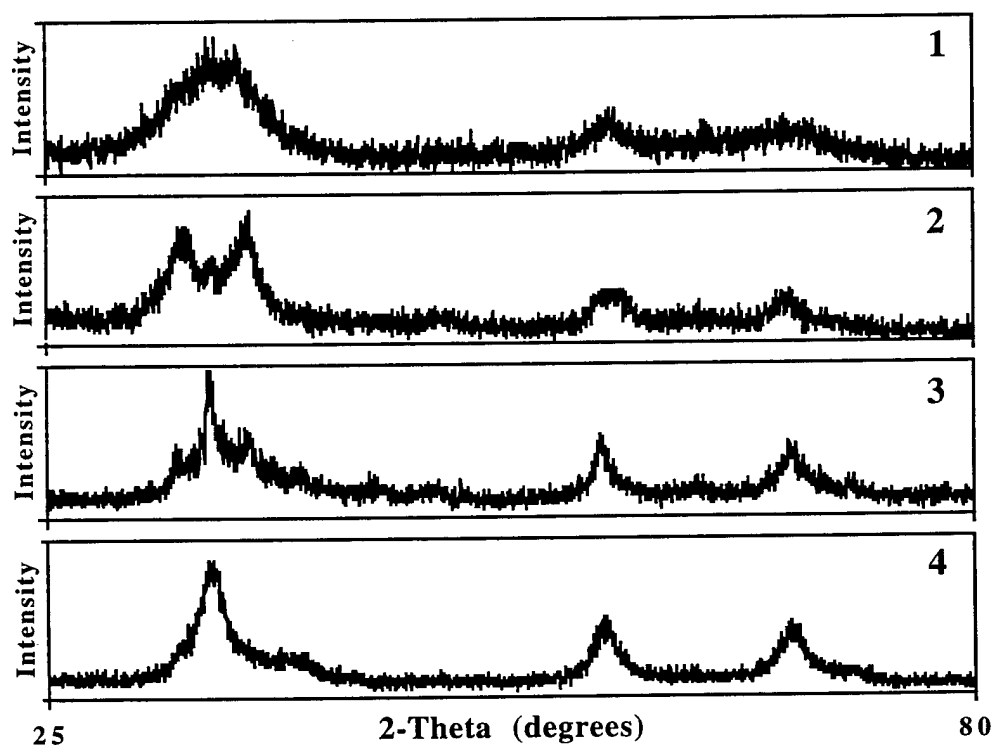


Figure 2. XRD powder patterns for gallazane derived GaN. Pyrolysis conditions for: **1** - 600 °C, vacuum, 4 h; **2** - 600 °C, NH₃, 3 h; **3** - 600 °C, NH₃, 3 h; **4** - 600 °C, NH₃, 4 h.

Samples **2-5** result from almost identical pyrolysis conditions but display dramatically contrasting diffractograms. For example, the pattern for sample **2** fits well with the pattern for pure hexagonal GaN (JCPDS file 2-1078). On the other hand, the pattern for sample **4** matches reasonably well the theoretically calculated pattern for cubic GaN [2b], and samples **3** and **5** show an intermediate phase form comparable with the previously discussed sample **D**. The pyrolysis

conditions being virtually the same, other factor(s) in the gallazane precursor route must be responsible for the observed span in the GaN varieties. We believe that it is the nature of the precursors utilized in this system. As mentioned above, the gallazane as well as the polymeric solids obtained from it are all thermally unstable. It is apparently this feature, which is the function of the preparative factors such as the type of the polymeric precursor, handling times, and extent of evacuation, that imprints in the chemical character of the precursors and, upon pyrolysis, is further transformed into the specific crystalline variety. For example, the elemental analyses indicate that there is a tendency for gallium enrichment in the final materials (see EA for samples 3 and 5).

Representative room-temperature PL spectra for GaN prepared by pyrolysis of the gallium imide are illustrated in Figure 3. Such measurements are useful in this context as a means of assessing the presence of defect or non-radiative sites in the solid(s). However, spectroscopic distinctions between cubic and hexagonal forms are possible only *via* analysis of excitonic luminescence at low temperature. Nanophase GaN prepared by the pyrolysis of the gallium imide precursor at or above a temperature of 500 °C either *in vacuo* or in an NH₃ atmosphere will typically produce samples exhibiting defect emission and a relatively low quantum efficiency. This is exemplified by the PL spectrum of GaN prepared by the pyrolysis of gallium imide at 600 °C under vacuum (sample B). Such weak PL is attributed to the presence of non-radiative defects of either interfacial or interior character. However, much stronger PL (albeit defect emission) can be observed when the pyrolysis temperature is lowered or the reaction medium is changed.

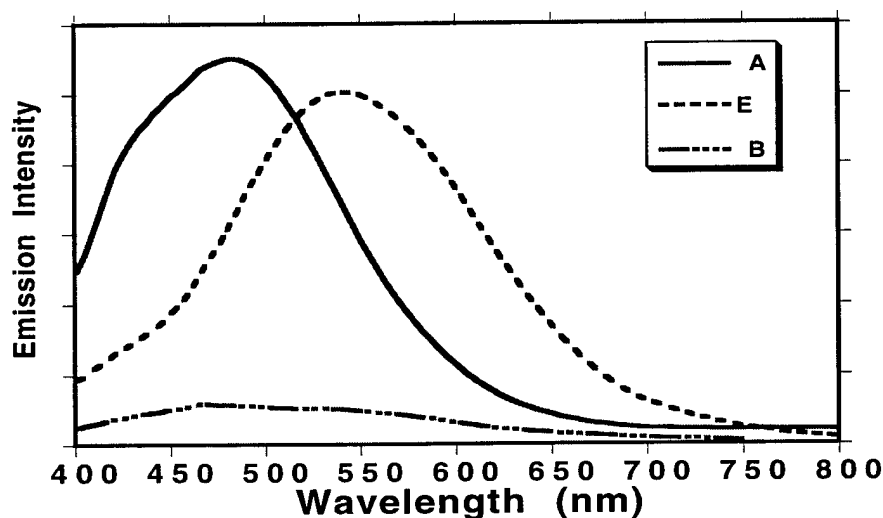


Figure 3. Room temperature PL spectra for GaN samples prepared by pyrolysis of gallium imide: **A** (solid line) - reflux in N,N,N',N'-tetramethyl-2,6-hexanediamine, bp 210 °C, 48 h; **B** (dot-dashed line) - 600 °C, vacuum, 3 h; **E** (dashed line) - 300 °C, NH₃, 4 h.

For example, when the gallium imide precursor is pyrolyzed in an ammonia atmosphere at a lower temperature of 300 °C, a more intense defect PL spectrum with a broad peak maximum near 560 nm is observed (sample E). It is found that a vacuum anneal of this type of GaN at 500 °C overnight results in diminution of the intensity of this band, consistent with its characterization as defect luminescence. Carrying out the reaction in a liquid phase *via* pyrolysis of the gallium imide in the relatively high boiling amine solvent N,N,N',N'-tetramethyl-2,6-hexanediamine (bp 210 °C) for 48 hours yields GaN which emits strongly in the blue region of the spectrum (430-480 nm); however, some of this broad blue light originates from the pyrolyzed amine coating present on the semiconductor surface (sample A).

Given the presence of defects in many of these as-prepared GaN nanophase materials, finding a suitable physico-chemical method to eliminate the defects and produce blue band-edge luminescent GaN is mandatory. Previous accounts regarding bulk epitaxially grown GaN have demonstrated that HF and HNO₃ have some utility as etchants for removing oxide and

hydrocarbon impurities from GaN [8]. Hence we decided to evaluate the efficacy of these acids on the photoluminescence of nanophase GaN prepared by the routes described in this paper. For these experiments, gallazane derived GaN sample 4 (pyrolysis at 600 °C, NH₃, 4 h) was exposed to solutions of 35% HNO₃ or 48% HF. It is found that a very brief etch (20 seconds) using 48% HF followed by a water and an ethanol wash yields GaN exhibiting an intense blue-emitting PL spectrum (Figure 4).

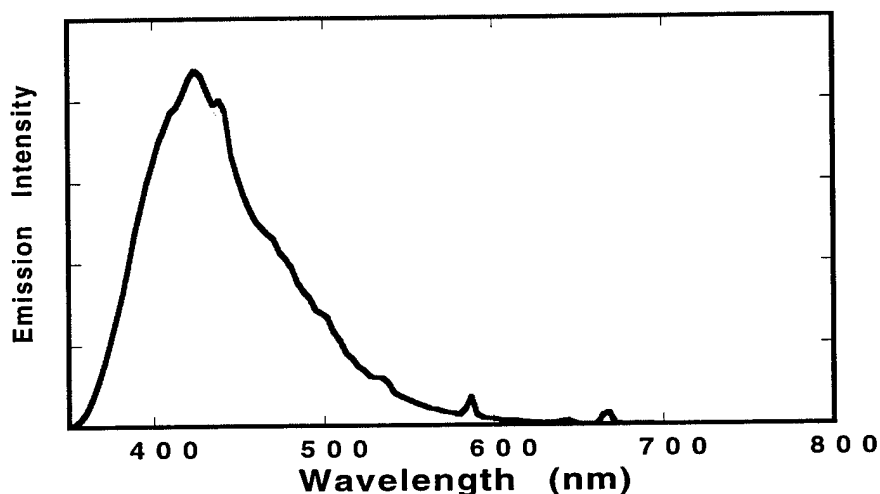


Figure 4. Room temperature PL spectrum of gallazane derived GaN sample 4 exposed to a brief HF etch.

At present, details of the mechanism of improved luminescence for these GaN nanophase samples are not clear. However, it is likely that the removal of oxide-related non-radiative centers by the acid at the nanoparticle surface plays a key role in these observations.

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800 N. Quincy Street
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